

Ash Removal from a Sample Coal by Flotation Using Rhamnolipid Biosurfactants

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Abstract- Synthetic chemicals from petroleum origins are now coming to a turning point on their hazardous problems to living organisms. In this regard, biosurfactants are well acknowledged as potential constitute for their chemical counter-part due to structural similarities. In this study, the surface activity and frothability of the rhamnolipid product produced from a *Pseudomonas aeruginosa* strain were measured. Then, frothing behavior of rhamnolipid as a frothing reagent to a sample coal flotation was evaluated. Rhamnolipid was found to have high surface activity and frothing capacity compared to pine oil as conventional frother in coal flotation practices, due to its higher molecular weight and presence of multiple oxygenated groups in its structure. The overall performance of sample coal flotation decreased as rhamnolipid concentration was increased. The decrease in flotation efficiency can be ascribed to probable depressing action of rhamnolipid to coal particles.

Keywords- *Pseudomonas aeruginosa*; *Biosurfactant*; *Frothability*; *Froth Flotation*; *Coal*

I. INTRODUCTION

Froth flotation, as a process in which fine coal particles are separated selectively from associated minerals in water slurries, by attachment to rising air bubbles, has a long history in coal beneficiation history. There are three main phases in a coal flotation process, i.e. coal particles, water and air bubbles that are affected by several physical and chemical parameters. The type and concentration of frother and collector are the key chemical factors in determining the overall separation performance, the clean coal recovery and purity (grade) of the concentrate. To qualitatively summarize the role of the frother in industrial flotation process it can be said that the presence of a surface-active agent (frother) leads to an enhance formation of a froth; an increase in the dispersion of air in the flotation cell, a reduction in the rate at which the bubbles rise to the surface and a reduction in the coalescence of individual bubbles within the flotation pulp which all influence the overall rate and performance of flotation practice in a significant manner [1-4].

With increasing environmental awareness and emphasis on a sustainable society in harmony with the global environment, during the recent years, natural surfactants produced by living cells are getting much more attention as compared to the synthetic chemical surfactants. Among the natural surfactants, those produced by microbial origin, known as microbial surfactants or biosurfactants are the most promising [5]. This is due to the advantages of biosurfactants over their chemical counterparts including lower toxicity, better environmental compatibility, biodegradability, and effectiveness in a wide range of temperatures and pH. Additionally, their production by renewable resources provides further impetus for serious consideration of

biological surfactants as possible alternatives of the commonly used industrial chemicals [6]. Among the various species of biosurfactants much work has been done on rhamnose containing microbial surfactants produced by *Pseudomonas aeruginosa* strains. This ubiquitous environmental bacterium can be isolated from many different habitats including water, soil and plants. Numerous articles consider the process of microbial cultivation of rhamnolipid type biosurfactants on different substrates and their potential application [7-26].

The possible application of rhamnolipid biosurfactants in ash removal from coal was first investigated by Fazaelpoor et al. [27] who evaluated frothing applicability of a rhamnolipid product (purity < 50%) to coal flotation. However, the low purity of the applied rhamnolipid challenges their results. More recently, Khoshdast et al. [28] used a highly pure (> 97%) rhamnolipid biosurfactant in coal flotation and showed that the separation process is inefficient in the presence of rhamnolipid biosurfactant.

In the present study, the effect of a concentrated rhamnolipid biosurfactant on the ash removal performance of a coal sample by froth flotation is investigated with a reference to coal/rhamnolipid interaction and the process selectivity evaluation.

II. MATERIALS AND METHODS

A. Bacterial Strain, Culture Medium and Biosurfactant Production

A pure strain of *Pseudomonas aeruginosa* MA01 (accession no. GQ478669) was obtained from microbial bank of the National Institute of Genetic Engineering and Biotechnology (NIGEB), Iran was used in the present work for biosurfactant production. The bacterial strain was pre-cultured on nutrient broth over a night at 30°C and 200 rpm before being inoculated into a designated medium for batch fermentation to produce the biosurfactant. The medium used for batch fermentation consisted of NaNO₃ (3 g/l), MgSO₄·7H₂O (0.25 g/l), KH₂PO₄ (0.25 g/l), yeast extract (1 g/l). The foregoing medium was supplemented with 40 ml soybean oil as carbon sources. The batch culture was incubated at 30°C and 200 rpm with a typical cultivation time of 10 days.

Rhamnolipid production was carried out in 2-l Erlenmeyer flasks containing 700-ml aliquots of the fermentation medium under test. After fermentation finished, the cell suspension was centrifuged (Sigma model 6-16K, Germany) 16,000 ×g for 15 min at 4°C to prepare the cell-free supernatant (CFS). The CFS was acidified with 1 N HCl

to pH 2 and left overnight at 4°C to precipitate rhamnolipids. The precipitate was harvested by centrifugation at 18,000 ×g for 50 min at 4°C. Afterwards, the precipitate was washed by acidified (pH 2) distilled water and centrifuged at 18,000 ×g for 10 min at 4°C. Rhamnolipid was mixed with an equal volume of ethyl acetate, vigorously shaken for 10 min, and centrifuged at 18,000 ×g for 15 min at 4°C. Finally, the pooled organic phase was transferred to a new vessel and evaporated under vacuum (Buchi model R-200, Germany) at 35°C [29,30]. After solvent evaporation, about 9 g of a viscous honey-colored biosurfactant product was extracted per liter of the culture medium. Crude biosurfactant was then analyzed using Fourier-transform infrared (FTIR) spectroscopy and Electrospray ionization mass spectrometry (ES-MS) methods for its purity and composition determination. Results showed that the rhamnolipid product was of 98% purity and composed of two types of commonly found rhamnolipids, i.e. $R_1C_{10}C_{10}$ and $R_2C_{10}C_{10}$ (Fig. 1, for $n=6$), with a 0.5:0.5 molar ratio [31].

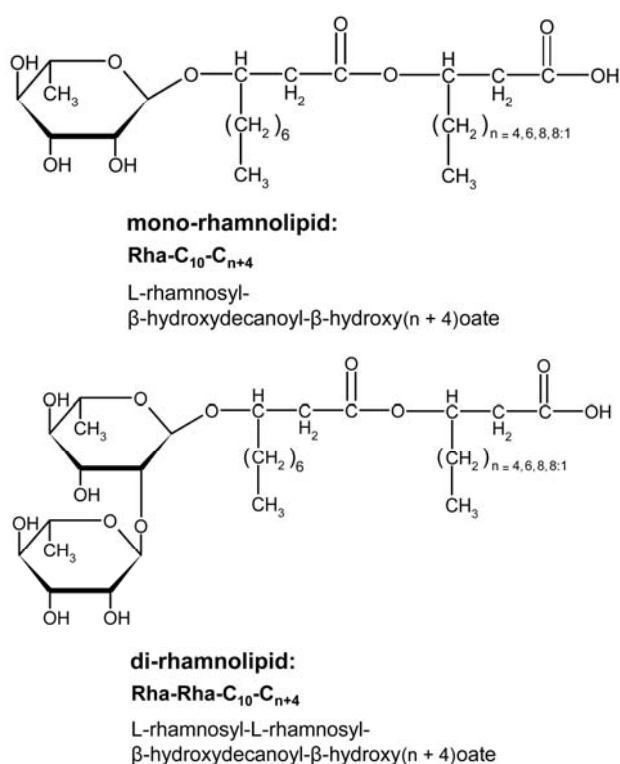


Fig. 1 The structure of rhamnolipid biosurfactants

B. Surface Tension Measurement

Surface tension measurements for rhamnolipid polluted solutions were conducted using a *du Nouy* ring tensiometer (Lauda model TD1-C, Germany). All the solutions were prepared at room temperature and stirred for 5 min before the measurements. To measure pine oil surface activity, 10 ml of frother (without further dilution) was placed into a clean glass beaker (50 ml) specific for the surface tensiometer (Lauda). Each measurement was replicated three times and an averaged value was reported.

C. Dynamic Frothability Index Measurement

The experiment for frothability characterization was performed in a glass cylinder with the height of 100 cm and the inner diameter of 5 cm. A sintered glass frit (10–15 μ m,

porosity 4) was at the bottom of the column. To determine frothability, the variation in froth volume was measured as a function of air flow rate. Results were then used to calculate dynamic frothability index (DFI) of surfactants. Three repetitive runs were made for each surfactant concentration level and airflow rate. All the experiment was conducted at a room temperature of 25±2°C. Froth characteristics of the biosurfactant were compared with those of pine oil as a conventional frother.

D. Coal Flotation Tests

A representative minus 0.5 mm size semi bituminous flotation feed coal sample, from Zarand Coal Washing Plant, Iran, was taken in this investigation. All the tests were carried out at a solid content of 8% by weight in a D-12 Denver flotation machine equipped with a 2 L cell. The pulp level was maintained constant by adding water as required. All the tests were carried out at natural pH (7±0.2) using pine oil (operating frother), rhamnolipid biosurfactant, and mixtures of them at different dosages and ratios. High-speed gas oil was used as a conventional collector (1.5 l/t).

Appropriate amounts of coal samples were mixed with 1 L of tap water in the flotation cell for 5 min to ensure that all coal particles were completely wetted. Following conditioning, the cell was filled with water to a set level and impeller speed was adjusted to 1000 rpm. Then gas oil (collector) was added and conditioned for 2 min. Four different frother systems, i.e. pine oil to rhamnolipid ratios, were considered to evaluate effect of rhamnolipid addition on coal de-ashing behavior. The added frother(s) was conditioned with the pulp for another minute. The concentrates were collected after 10, 20, 30, 45, 75, 105, 135, and 210 s of flotation in separate trays (previously weighed). The froth attached to the scrapper was washed-off every 10 s when collecting the concentrates. The concentrates and tailings were dried in an oven at 60°C for 24 h.

Ash analyses were carried out according to ASTM D 3174-73 Standard showed that the bulk sample contains 21.76% ash. Before ash analysis, products were first allowed to settle, decanted and then dried at 60°C.

III. RESULTS AND DISCUSSION

A. Surface Tension Measurement

The equilibrium surface tension for the studied surfactants is shown in Fig. 2. The surface tension decreases with increasing frother concentration as molecules adsorb at the liquid–air interface.

The plots clearly show that rhamnolipid decreases the surface tension more sharply compared to pine oil frother. It indicates that rhamnolipid is more surface active than pine oil. Rhamnolipid has higher molecular weight (577 g/mol) than pine oil (140 g/mol) and this higher molecular weight may lead to higher surface activity of rhamnolipid. Pine oils commercially used in mineral flotation industries are mainly composed of α -terpineol ($C_{10}H_{17}OH$) containing only one –OH group which interact with water molecules to form an oriented monolayer at the surface. Whereas, rhamnolipid contains several oxygenated units in their molecular chain which can interact with water molecules through hydrogen

bonding, causing the molecules to tend to lie at the surface, providing much closer packed cohesive film than pine oil [31].

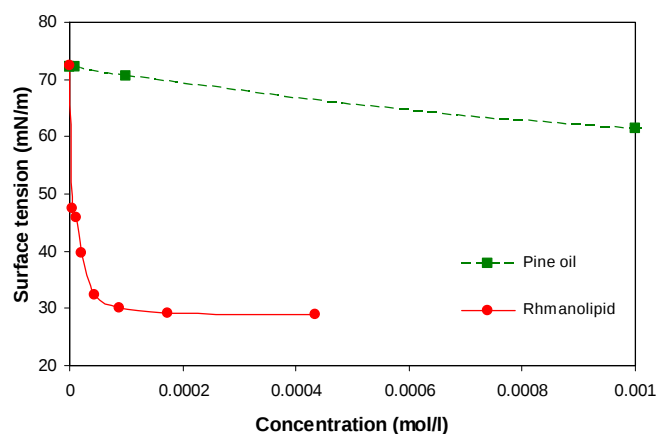


Fig. 2 Variation of surface tension with frother concentration

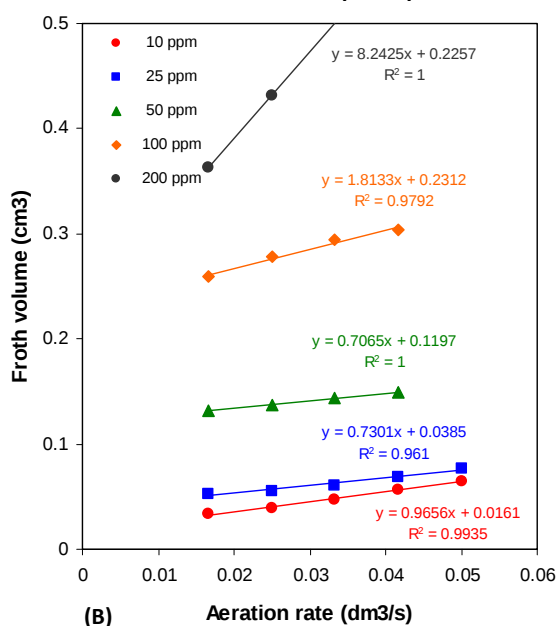
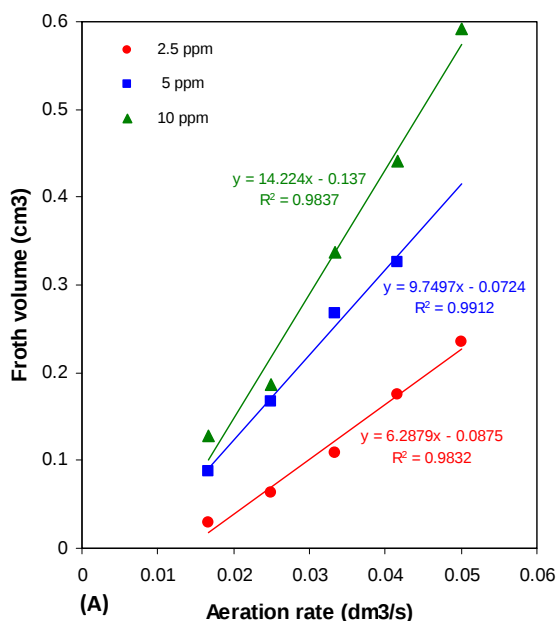


Fig. 3 Graphical determination of retention time for rhmanolipid biosurfactant (a) and pine oil (b)

B. Dynamic Frothability Index Measurement

Frothability reflects the ability of a frother to produce a froth phase in both terms of froth capacity and persistency. The DFI stands for dynamic frothability index and is determined from the values of retention time, rt , which is the slope of the linear part of the dependence of the total gas volume contained in the system, V , on the volumetric gas flow rate, Q [32]:

$$rt = \frac{\Delta V}{\Delta Q} \quad (1)$$

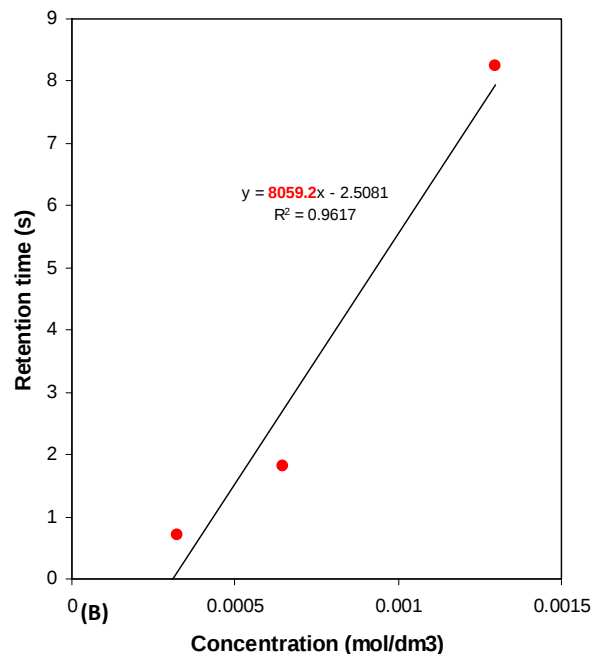
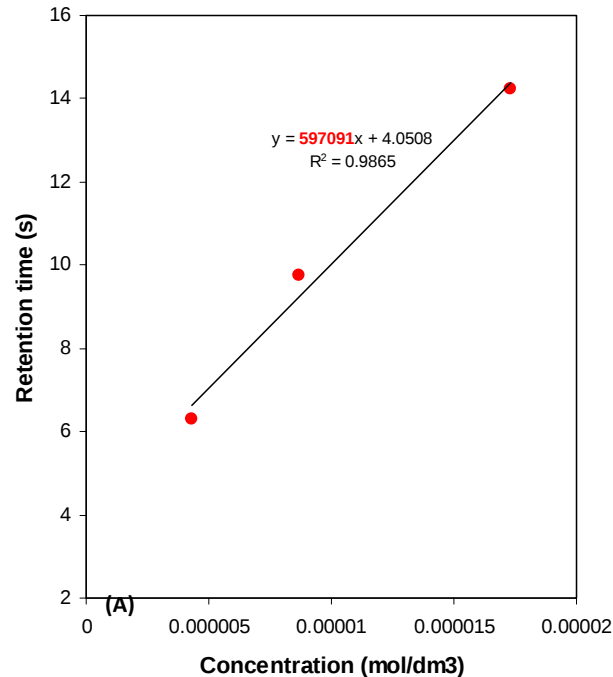


Fig. 4 Graphical determination of DFI for rhmanolipid biosurfactant (a) and pine oil (b)

This slope increases with increasing frother concentration and DFI is defined as the limiting slope of the rt versus concentration for $c \rightarrow 0$:

$$DFI = \left(\frac{\partial \tau}{\partial c} \right)_{c=0} \quad (2)$$

Steps on calculating the retention time and DFI are graphically shown in Figs. 3 and 4, respectively. From Fig. 3, the DFI for rhamnolipid is obtained 597,091 s.dm³/mol. Similarly, the DFI for pine oil was obtained 8,059.2 s.dm³/mol. Larger DFI values indicate more stable foam, in which bubbles do not easily coalesce [2]. Thus, larger DFI value of rhamnolipid than pine oil indicates that rhamnolipid can act as a powerful frother increasing the rate of flotation process. In comparison, pine oil has higher selectivity and improves the flotation performance of fines and/or particles with low density [33].

C. Coal Flotation Tests

A series of flotation experiments were carried out to compare single and mixed systems for pine oil and rhamnolipid. The surfactants were compared under like to like operating conditions (1.5 l/t collector, 3 l/min air flow rate, 8% solid content in feed and 1000 rpm impeller speed) based on the following criteria:

- Yield and ash content (performance),
- Yield vs. ash content (selectivity),
- Rate constants (kinetics).

The metallurgical parameters for combustible material (clean coal) and non-combustible bearing mineral (ash) were calculated through following general equations [4]:

$$\text{Yield} = (\text{Tailing ash} - \text{Feed ash}) \times 100 / (\text{Tailing ash} - \text{Froth ash}) \quad (3)$$

$$\text{Separation efficiency} = \frac{\text{Combustible recovery}}{\text{Ash recovery}} \quad (4)$$

$$\text{Combustible matter recovery} = \frac{\text{Yield} \times (100 - \text{froth ash})}{(100 - \text{Feed ash})} \quad (5)$$

$$\text{Ash matter recovery} = \text{Yield} \times \text{Froth ash} / \text{Feed ash} \quad (6)$$

1) Yield and Ash Content of Products (Performance)

Fig. 5 shows the effect of rhamnolipid addition on coal yield and ash content of concentrates. As seen, coal yield

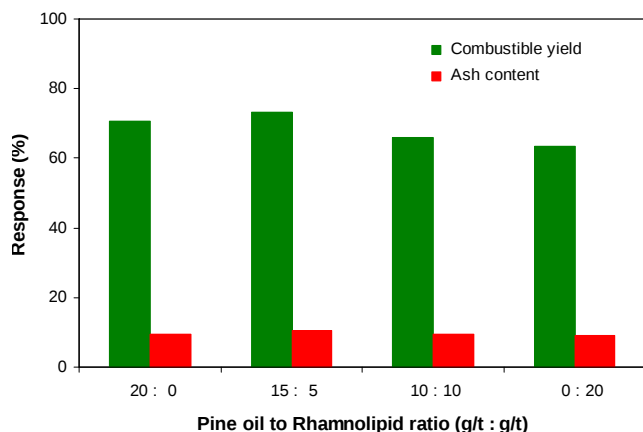


Fig. 5 Effect of rhamnolipid addition on metallurgical responses of coal flotation

decreases by rhamnolipid concentration whereas ash content variation is negligible. Thus, it could be concluded that rhamnolipid addition negatively influences the process performance of coal de-ashing by froth flotation. This can be attributed to probable depressing effect of rhamnolipid molecules on coal particles either in the absence and/or the presence of gas oil as collector, as schematically illustrated in Figs. 6(a) and 6(b), respectively.

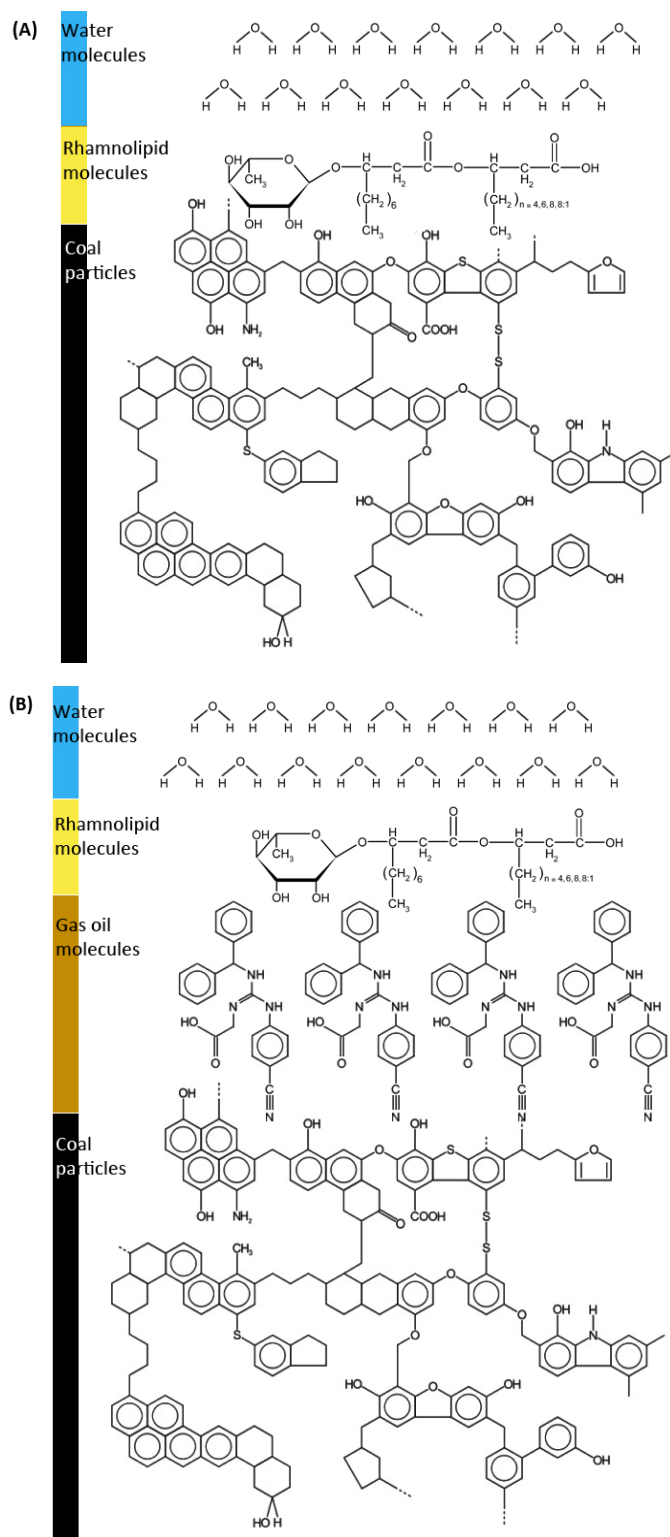


Fig. 6 Potential mechanism for coal particle depression by rhamnolipid molecules (mono-type in this figure), in the absence (a) and the presence (b) of gas oil as collector

As shown in Fig. 6(a), rhamnolipid molecules comprise two long hydrocarbon chains that can interact with aromatic rings at the surface of coal particles through van der Waals bonding. This may lead rhamnolipid molecules to arrange at the coal surface such that the carboxyl groups are oriented into the water phase. Oxygenated functions are then interact with water molecules through strong hydrogen bonds decreasing the natural hydrophobicity of coal particles and consequently, depress them. Some hydrogen bonding between the hydroxyl groups at rhamnolipid side toward the coal surface and surface hydroxyl groups of coal may also form that will improve rhamnolipid/coal attachment stability.

Gas oil is the most commonly used non-ionic collector in coal flotation practices. Gas oil will interact with coal particles by van der Waals bonding and to some extent, with a number of hydrogen bonding (Fig. 6(b)). The hydrocarbon rings of gas oil molecules are then aligned toward water phase and significantly increase the hydrophobicity and floatability of coal particles. As shown in Fig. 6(b), the hydrocarbon chains of rhamnolipid can interact with gas oil rings through multiple van der Waals linkages. Rhamnolipid molecules will then make hydrogen bonding with water molecules and depress the coal particles. The interaction between rhamnolipid molecules and non-combustible components (ash mineral) could be neglected since the variation in froth ash content is not significant.

2) Yield vs. Ash Content (Selectivity)

The clean coal yield versus ash content plots give useful information on the selectivity of a process [4,34]. The representative lines positioned at higher levels in the graph suggest better selectivity for corresponding processes. Fig. 7 shows the selectivity for different frother systems. It reflects that the selectivity of the process increases by pine oil concentration. This is in agreement with the conclusion drawn from the DFI calculation.

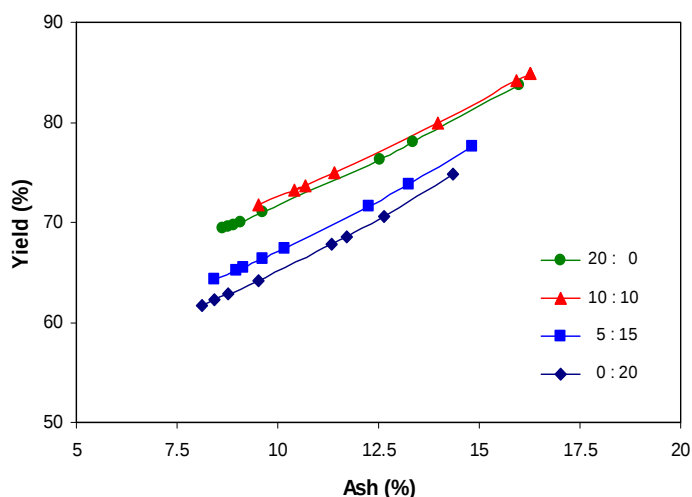


Fig. 7 Comparison of frother systems (pine oil : rhamnolipid ratio) on the basis of selectivity (yield-ash relationship)

3) Rate Constants (Kinetics)

Based on direct experimental observations in both batch laboratory and continuous large scale flotation cells, the use of a frother significantly increases first, the possibility of a particle-bubble contact, and second, the efficiency of sticking

after such a contact. Thus, a major role of a frother is to significantly increase the rate of flotation [35]. The flotation process can be explained as a rate process, since rate of recovery of particles is proportional to the concentration of floatable particles remaining in the pulp at time t . The simple first order rate equations for batch flotation can be written as:

$$r = -\frac{dw}{dt} = kw \quad (7)$$

$$\ln(1 - R) = -kt \quad (8)$$

where r is flotation rate, w weight of floatable mineral remaining in the pulp at time t , k first order rate constant (for combustible material or ash mineral), t cumulative flotation time, and R the recovery of the component (combustible material or ash mineral) at time t . When the curve is fitted to a set of experimental data pairs of R of the component and t , the parameter k is numerically determined with appropriate statistical confidence regions.

In Fig. 8, the flotation rate constants of clean coal and ash materials for studied frother systems are shown. As seen, the rate constants for both coal and ash components follow an increasing trend as rhamnolipid concentration increases. This implies that rhamnolipid biosurfactant acts as a powerful frother which was indicated by DFI calculation.

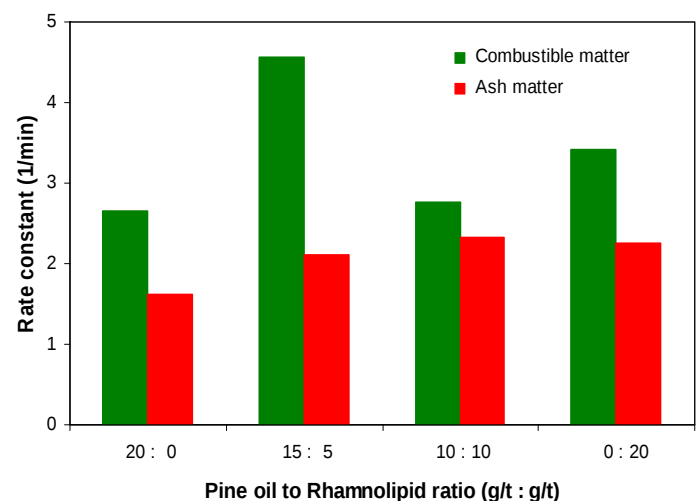


Fig. 8 Comparison of frother systems on the basis of kinetics (flotation rate constant)

IV. CONCLUSIONS

The surface and frothing properties of a rhamnolipid biosurfactant produced from *Pseudomonas aeruginosa* strain were characterized. Results showed that rhamnolipid is of high surface activity and frothing power as indicated by dynamic frothability measurement. The use of the rhamnolipid product in a sample coal flotation as sole frother of the process or in mixed with pine oil, a commonly used frother in coal processing practices, demonstrated that rhamnolipid has could negatively influence the overall flotation performance. This was ascribed to the potential depression effect of rhamnolipid on coal particles in the absence and the presence of gas oil as collector. The yield/ash correlation and kinetics studies showed that rhamnolipid biosurfactant has low selectivity and high frothability compared to pine oil. Further fundamental studies are

required to describe the real interaction between rhamnolipid, coal surfaces and gas oil molecules.

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